

- The resultant dispersion after allowing to settle for 2 h, was used to coat the inner wall of the thoroughly cleaned silica glass tubes with inner diameter of 17.8 mm. The outer wall of the tubes were properly masked with a suitable substance (parafilm).
- Coating was performed by dipping the silica glass tubes with a speed of 10 cm/min into the dispersion and lifting the same tube from the above dispersions with the same speed.
- The coated tubes were dried in air at 100°C for 1h.
- Oxidation at temperatures of 610°, 700°, 825° and 950°C with 2 passes of the burner at each temperature maintaining a constant He/O<sub>2</sub> ratio of 1:5 .
- The dehydration was carried out at a temperature of 1000°C with a Cl<sub>2</sub>: O<sub>2</sub> ratio of 2 : 1 for a period of one hour.
- For sintering the temperature was increased in 4 steps up to 1220°C, each step consisting of 2 passes of the burner. GeCl<sub>4</sub> was added in controlled quantities during this stage with the input oxygen with two passes at 1220°C in order to adjust the NA of the preform / fibre.
- The tube was further heated to increase the temperature stepwise to 1600°C for complete sintering of the Ge, Eu & Al containing coated layer. During sintering O<sub>2</sub> and He flow was in the ratio of 4:1.
- The collapsing was done in 3 steps in the usual manner with positive oxygen pressure of 4 psi inside the tube to avoid any deformation in shape or geometry and excessive evaporation of GeO<sub>2</sub> or other oxides from the core.
- The core:clad ratio before overcladding was 7.2:125. The NA measured in the fibre was  $0.14 \pm 0.01$  .
- The presence of Eu in the core was established from the characteristic fluorescence at 624 nm in the preform sample when excited at 392 nm.
- Spectral attenuation was measured in the fibre from 400 nm to 800 nm.

#### EXAMPLE 4

##### Tb-doped fibre

- Amorphous silica microspheres synthesized by hydrolysis of tetraethoxyorthosilicate (Stober method) were dispersed in a solution of terbium nitrate (kept in an ice bath) in a proportion of 98.5 mol% SiO<sub>2</sub> and 1.5 mol% Tb<sub>2</sub>O<sub>3</sub> under sonication followed by the addition of aqueous ammonia by known process. The resulting product was washed with water followed by centrifugation and drying under vacuum.
- A stable dispersion of composition 95.9SiO<sub>2</sub>: 2GeO<sub>2</sub>: 2Al<sub>2</sub>O<sub>3</sub>: 0.1 Tb<sub>2</sub>O<sub>3</sub> (in equivalent oxide mol%) was prepared for the application of coating to the inner wall of high purity clear fused silica glass tubes.
- From the terbium oxide (Tb<sub>2</sub>O<sub>3</sub>) coated silica powders with 98.5 mol% SiO<sub>2</sub> and 1.5 mol% Tb<sub>2</sub>O<sub>3</sub>, a silica sol of composition of 95.9 equivalent mol% of SiO<sub>2</sub> and 0.1 equivalent mol% of Tb<sub>2</sub>O<sub>3</sub> was prepared by diluting with a silica sol containing the desired amount of silicon tetraethoxide (TEOS).
- Silica-germania sol containing 2 equivalent oxide mol% of germanium ethoxide [Ge(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>] was prepared through the hydrolysis of TEOS and [Ge(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>] with water and hydrochloric acid in presence of a mixed solvent of propan-1-ol and butan-2-ol. pH of the above sol was 1.2 ± 0.05 .
- 2 equivalent oxide mol% of [Al(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O] and 0.1 equivalent mol% of Tb<sub>2</sub>O<sub>3</sub> through Tb<sub>2</sub>O<sub>3</sub> coated SiO<sub>2</sub> powders (after baking at 100°C for 1h) were dispersed in the above silica sol under sonication (26 kHz) for 80 mins.
- The resultant dispersion after allowing to settle for 2 h, was used to coat the inner wall of the thoroughly cleaned silica glass tubes with inner diameter of 20.1 mm. The outer wall of the tubes were properly masked with a suitable substance (parafilm).
- Coating was performed by dipping the silica glass tubes with a speed of 10 cm/min into the dispersion and lifting the same tube from the above dispersions with the same speed.
- The coated tubes were dried in air at 150°C for 0.5h.

- Oxidation at temperatures of 720°, 825° and 950°C with 2 passes of the burner at each temperature maintaining a constant He/O<sub>2</sub> ratio of 1:6.
- The dehydration was carried out at a temperature of 950°C with a Cl<sub>2</sub>: O<sub>2</sub> ratio of 2.5 : 1 for a period of one hour.
- For sintering the temperature was increased in 3 steps up to 1200°C, each step consisting of 2 passes of the burner. GeCl<sub>4</sub> was added in controlled quantities with the input oxygen during the passes at 1200°C in order to adjust the NA of the preform / fibre.
- The tube was further heated to increase the temperature stepwise to 1650°C for complete sintering of the Ge, Tb & Al containing coated layer. During sintering O<sub>2</sub> and He flow was in the ratio of 4:1.
- The collapsing was done in 4 steps in the usual manner with positive oxygen pressure of 4 psi inside the tube to avoid any deformation in shape or geometry and excessive evaporation of GeO<sub>2</sub> or other oxides from the core.
- Overcladding was done to reduce the core:clad ratio to 5:125. The NA measured in the fibre was  $0.11 \pm 0.01$ .
- The preform sample was excited at 355 nm and 400 nm and characteristic fluorescence of Tb was measured at 533 nm and 580 nm.
- Spectral attenuation was measured in the fibre from 400 nm to 800 nm

**Comparison of the characteristics of RE-doped fibre.fabricated at CGCRI using RE-coated silica nano particles w.r.t. commercially available fibres for special applications**

Parameters	Commercial fibre	CGCRI fibre
Clad composition	SiO <sub>2</sub> or SiO <sub>2</sub> - P <sub>2</sub> O <sub>5</sub> -F	Same
Core composition	SiO <sub>2</sub> +GeO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> + Er <sub>2</sub> O <sub>3</sub> (+P <sub>2</sub> O <sub>5</sub> )	Same
Numerical aperture	0.15 - 0.30	0.10 - 0.30
Cut-off wavelength	850 - 1450 nm	850 - 1450 nm
RE-ion concentration	50 - 2000 ppm	50 - 4000 ppm
Fibre diameter	125 ± 1 µm	125 ± 1 µm
Coating diameter	245 µm (nominal)	245 µm
Mode Field Diameter	3.5 - 8.0 µm	3.0 - 8.0 µm